

Operating the Microprobe

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Effective Date: 3-16-92

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Operating the Microprobe

1.0 PURPOSE

This procedure provides instructions for the operation of the Cameca MBX Electron Microprobe and Tracor Northern (TN) Series II X-ray Analyzer.

2.0 SCOPE

2.1 Applicability

This procedure applies to electron microprobe analyses of geologic specimens and other materials documented as Los Alamos Environmental Restoration (ER) Program samples using the Cameca MBX Electron Microprobe and TN Series II X-ray Analyzer.

2.2 Training

Training for operators consists of reading and applying this procedure and the instruction manuals for the Camera MBX Microprobe, SANDIA TASK8 Automation System and Tracor Northern Series II X-ray Analyzer. Additionally, operators receive training through hands-on operation of the equipment under the supervision of the machine custodian. The training is recorded by the machine custodian in the ER EMP System Logbook and in the operator's personnel file.

3.0 DEFINITIONS AND/OR ABBREVIATIONS

- A. TN: Tracor Northern (now Noran Instruments, Inc.)
- B. EDS: Energy Dispersive Spectrometer
- C. WDS: Wavelength Dispersive System
- D. EMP: Electron Microprobe
- E. EMP System: Combined Cameca MBX Electron Microprobe & Series II X-ray Analyzer System.
- F. SANDIA TASK8: A Subroutined Electron Microprobe Automation System.
- G. BA: An empirical matrix correction program used specifically for oxide minerals. Refer to B85: A Bence-Albee Oxide Analysis Program with Mineral Code Capabilities.
- H. PRZ: A quantitative matrix correction technique based on the depth distribution of X-ray production. Refer to PRZ85: A Subroutined Quantitative Analysis Correction Procedure Using the Bastin q(rz) Technique

4.0 BACKGROUND AND/OR CAUTIONS

The basic principle of the Electron Microprobe is the generation of a highly focused beam of electrons which strike and interact with the constituent elements of the target specimen. These interactions produce characteristic x-rays which are then detected by wavelength and energy dispersive spectrometers. The intensity of each x-ray is proportional to the concentration of the element present. Through comparison of the corrected X-ray intensity from each element to those from specimens of known concentration, unknown compositions may be quantitatively determined.

It is the responsibility of the machine custodian to perform the routine maintenance of the system including: system software and electronically-stored system back-ups; calibration of EDS gain; EDS resolution check; Faraday Cup check; instruction of the operators in the use of the system; and certification of operators for this procedure. The machine custodian may delegate responsibilities documenting any delegation in the ER EMP System logbooks.

Operators are responsible for calibration of semi-quantitative and quantitative analytical routines used in their particular data acquisitions. The Operator must ascertain that all EMP systems are operational before an investigator begins a microanalysis session. The Operator determines acceptance or rejection of data generated by the Electron Microprobe analysis routines. The Operator also is responsible for the proper storage and disposition of hard copy and magnetic media data records, and disposition and/or storage of samples and materials. Acceptance criteria will be entered on the Daily Activity Log (SOP-01.04). Deviations from this procedure must be documented by the responsible Investigator on the Daily Activity Log.

Malfunction of the EMP System should be readily detectable by operators during analysis of standards. If the operator is uncertain, he/she may consult with the machine custodian. If the EMP vacuum system is not sufficient to allow operation, consult with the machine custodian.

Normal interior building temperature and humidity are acceptable for the operation of the EMP System. Cooling water for the EMP diffusion vacuum pump is supplied by a chilled water system maintained in the range of 40° to 70°F. Ambient air temperature for the EMP System should range between 60° and 80°F. Operators should take extra precaution to ensure the instrument maintains calibration by checking standards more frequently when environmental conditions are out of range.

5.0 EQUIPMENT

- The Electron Microprobe is a Cameca Model MBX (purchase date 1976). Refer to the Cameca Operating Manuals for description of this system.
- The X-Ray Analyzer is a Tracor Northern Series II X-Ray Analyzer (purchase date August 1988). Refer to the Tracor Northern Operator's Manual for this system.

- EMP System software is commercially acquired software that includes Sandia Task 8 and the TN Series II X-ray Analyzer Software. Both software packages operate under the "FLEX" operating system, manufactured by Noran Instruments (formerly Tracor Northern Instruments). Software upgrades are received as releases directly from the software manufacturers.

6.0 PROCEDURE

Certified standards should be used for calibration of quantitative x-ray analysis routines. Operators may use other standards for specific applications but must document the basis for usage of these standards on the Daily Activity Log.

Data consist of elemental information which is printed following each analysis. Paper hard copy contains the sample identifier cross referenced to the operator's ER sample number in their own ER notebook. Elemental information is also stored on magnetic media for retrieval at a later date.

6.1 Sample Preparation

Samples to be examined in the EMP System must be in a form or size that can be attached to an EMP sample mount and inserted into the EMP sample chamber. Before being placed in the EMP System, all non-conductive samples must be given an electrically conductive carbon coating. Conductive samples may not require coating. The operator will determine if a conductive coating is appropriate. Carbon coating procedures are obtained in the Instruction Manual, Ladd Vacuum Evaporator.

Sample identification and control are described in SOP-1.04, Sample Control and Documentation. If it is not practical to place entire ER sample identification number on the sample mount, an abbreviated number may be placed on the mount, but the abbreviated number must be keyed to the full number in the operator's ER notebook.

6.2 Microprobe Start-up

- 6.2.1. The microprobe is in Stand-by condition after shut down at the end of the previous work day.
- 6.2.2. Fill the liquid nitrogen cold trap. Refill it in approximately 30 minutes; then every four hours.
- 6.2.3. If it is not already on, turn on the TN Series II monitor power switch located on far lower right side of the monitor console, next to the contrast dial. Turn contrast up to acceptable level.

- 6.2.4. Turn on the TV camera adaptor located below the TV monitor. Turn on the TV monitor.
- 6.2.5. Turn on the Light Microscope Illuminator by pushing in the Illuminator button. Switch the toggle below it to High.
- 6.2.6. Position the white toggle switch located on the column above the stage indicators to reflected light. (Up is reflected light , middle is off, and down is transmitted light.)
- 6.2.7. Activate the Vacuum System by switching the toggle on the left front of the vacuum chassis to OPERATE.
- 6.2.8. Verify that the vacuum system has achieved suitable vacuum (less than 5×10^{-5} torr) as read from the secondary gauge position of the vacuum gauge and the Secondary Vacuum light is now on. If the secondary vacuum light does not come on consult with the machine custodian.
- 6.2.9. Verify that the program TASK8 is running by looking for the "TASK8": prompt at the bottom of the TN Series II monitor. If the prompt is correct proceed to step 6.4. If the prompt is not correct, type X 'TASK' (CR). If the prompt is now correct proceed to step 6.4, if it is still not correct continue in sequence and boot the computer.

6.3 Boot computer

- 6.3.1. Press the Reset switch located just below the Power switch on the TN Series II console (just to the right of diskette drives 5 and 6) and step through the following sequence of inputs.
 - (prompt on the printer) "28"
 - (prompt on the printer, sometimes absent)"START"
 - If the "28" prompt does not appear turn the computer power switch (above the Reset) Off; wait 5 minutes then turn it back on and start section 6.3.1 again. If it still does not boot correctly, consult with the machine custodian.
 - (type) DL (CR)
 - Wait for all the Program section softkeys to light up and then turn off. The TN logo will appear on the monitor screen.
 - Press the PROGRAM soft key (Program section of the soft-key board).
 - Select the LOAD FLEXTRAN option (option 0 will be highlighted when 0 is typed) and (CR).
 - (prompt) "SETUP COMPLETE", type (CR).
 - Press the RUN soft key
 - When the asterisk (*) prompt appears, (type) X 'START' (CR)
 - (prompt) "SKIP RAM DISK INITIALIZATION (Y/N)?"
 - (type) N (CR).

- 6.3.2. The system will take about two minutes to initialize the RAM and load libraries, system files, and general subroutines. It will then display these messages:
- "(Current date and time) Ram disk initialized"
 - "Position paper to the tear-off point and then hit the space bar". Do so.
 - "Use CONFIG file (CR or new #) 4000 ?"
 - (type)(CR). (Configuration file 4000 is set up to use an accelerating potential of 15 KeV. This is an optimum setting for most samples: sulfides however may require 20 KeV accelerating voltage. If another accelerating voltage is required consult with machine custodian. If the operator chooses to use another accelerating voltage, it must be documented in the operators ER notebook.)
- 6.3.3. The program will then display the current spectrometer and stage positions and ask "OK(Y/N)?". The current stage idle position (referred to as Standard Focus or Sta Fo) is posted beside the EMP and all spectrometers are parked at .50 (sine theta) overnight. Check the mechanical indicators on the stage and spectrometers and determine if the readouts are approximately the same as those listed. If the values are approximately the same, type (CR) and proceed to step 6.4. If the values are not similar, type N (CR) and continue with this sequence.
- 6.3.4. If a 'N (CR)' is entered, the computer will read out each one of the LED displays in the following order: X axis, Y axis, Z axis, spectrometer 1, spectrometer 2, spectrometer 3, and spectrometer 4. As each axis is listed, if the LED and the mechanical indicators agree then type a (CR). If they do not agree, type in the value as read from the mechanical indicators and then type a (CR).
- 6.3.5. After listing spectrometer 4, the computer will again type out all values and ask if "OK(Y/N)?". If the positions are now correct type a (CR) and continue to 6.4. If the positions are not correct type N (CR) and repeat the above step until they are correct.
- 6.3.6. The computer will now auto-load standard and element tables, look for the next free data file (on the operator's personal diskette) and then display the TASK8: prompt.

6.4 Saturating the Filament

- 6.4.1. Verify that the Secondary Vacuum LED is now on. If it is not, verify that the vacuum reading on the secondary gauge is less than 5×10^{-5} torr and press the silver reset button. The Secondary Vacuum LED should then come on. If the Secondary Vacuum LED does not come on, consult with machine custodian.

- 6.4.2. Type GET STA THO (CR). This will move the stage to the thorium standard and allow the Investigator to see the beam when it is turned on. Use the Z axis on the joystick to focus on the thorium standard.
 - 6.4.3. Type D B-1 (CR). This will release computer control of the filament circuit.
 - 6.4.4. Turn the Absorbed Current Range Selector to 3×10^{-8} ampere.
 - 6.4.5. Push in Filament Power Supply Button .
 - 6.4.6. Saturate the filament in the following manner: Slowly turn the Filament Temperature Knob clockwise to about 4 to 8. Watch the Absorbed Current Meter. The meter will read a fast rise in signal and then level out. The object is to peak the filament temperature just into the level out, or plateau area. Verify that the beam is visible on the thorium standard via the TV monitor. Select the largest square on the Raster Size Control Panel. Wait 15 minutes before proceeding to the next step; this will allow the beam to stabilize.
 - 6.4.7. Type D B 15 (CR). This will allow the computer to define the beam to fifteen nanoamperes, the most commonly used beam current and then blank the beam. However, the Investigator may choose the appropriate beam current and note it in the investigator's ER logbook.
 - 6.4.8. Type UNB (CR). This will unblank the defined beam and allow viewing.
 - 6.4.9. Press the Spot Raster Size Control button.
 - 6.4.10. Use Lens 3 to make the spot as small as possible.
 - 6.4.11. Use the Z axis on the joystick control to recheck the focus.
 - 6.4.12. Center the beam raster on the TV Monitor crosshairs by turning the two Optical Microscope Adjuster Knobs. The adjuster knobs are located at the base of the column in the 'vee' created by the column and spectrometers 1 and 4.
 - 6.4.13. Press the large square of the Raster Size Control.
 - 6.4.14. Type BL (CR). This will blank the beam
- 6.5 Quantitative Standard Analysis**
- 6.5.1. Insert the Certified Operator's personal disk into drive 5 (if not already there).

- 6.5.2. If a catalogue of the disk is desired, type CAT (CR). When prompted "Device #", enter the number of the disk drive (5) (CR), and (CR) again to list all of the disk contents. Note the last data file used and the next unused file on a piece of scratch paper.
- 6.5.3. The Certified Operator will select a Primary standard or group of standards from a standards list. The selected standard(s) should be similar chemically (e.g., carbonate, oxide, or silicate) to the unknown being analyzed. If a Primary standard is not available for a particular element the Certified Operator will select another standard and document the basis for usage in his/her ER notebook.
- 6.5.4. Type GET STA XXXX (CR), (where XXXX is the one to four character label of the selected standard). This will move the stage to the selected standard. Focus using the Z axis control on the joystick and if necessary manually drive onto the standard.
- 6.5.5. If analyzing oxide phases continue with this sequence. In this context, any mineral containing oxygen is considered to be an oxide phase. If analyzing non-oxide phases proceed to section 6.5.7.
- 6.5.6. Type Q BA (CR) for analysis of oxide phases. When prompted for the BA Definition File number, enter 110 for a generic Definition File that utilizes the Bence-Albee routine with mineral codes or enter the file number of the Certified Operators own previously set-up file. (Refer to "BA85: A Bence-Albee Oxide Analysis Routine with Mineral Code Capabilities" for information on setting up analysis-specific mineral codes. Refer to "Sandia Task8C" for information on setting up a Definition File for an analysis routine that does not access the mineral code routines). Proceed to section 6.6.8.
- 6.5.7. To perform analysis of non-oxide phases (e.g., the sulfides), the PRZ matrix correction program must be used. Type LOAD ELEMENT 112 (CR) to load an element table containing PRZ correction factors. Then type Q PRZ (CR). Refer to "SANDIA TASK8C" for information on how to set up a PRZ definition file and then, by following the prompts, create a definition file appropriate to analyze the desired elements. Continue in this sequence.
- 6.5.8. The file settings will be printed and the computer will ask if "OK(Y/N)?". Check to determine that the settings are appropriate. If the file is correct type a Y (CR). If the file does not meet the analysis requirements type a N (CR) and follow the prompts to correct the file. When prompted again "OK(Y/N)?" type a Y (CR) or repeat this step to further correct the file.
- 6.5.9. When prompted for the Setup file number, enter 1 (CR). The file settings for Page 1 of the Set-up file will be printed. Check that the printed settings meet the analysis requirements and then press the Cancel key. If they do not meet the analysis requirements, highlight the parameter to be changed and

type in the correct value. When correct, press the Cancel key. Follow the same procedure for Page 2 of the Set-up file and then when asked, "OK(Y/N)?" type Y (CR).

- 6.5.10. When prompted "SET-UP FILE #1 OK(Y/N)?" type Y (CR).
- 6.5.11. When prompted "BEAM SIZE, μM ?" type the beam size selected: spot = (approximately) 1 μm (diameter), small square = (approximately) 5 μm (diameter), large square = (approximately) 17 μm (diameter).
- 6.5.12. When prompted "PRINTER LABEL" type the ER sample number or an identifier for the sample or group of samples to be analyzed and (CR). The Certified Operator will cross-reference any analysis identifiers to the ER sample numbers in his/her ER notebook.
- 6.5.13. When prompted "ANALYST", the Certified Operator will type his or her complete last name and a (CR).
- 6.5.14. When prompted "RESERVE FILE SPACE FOR 48 PTS (NEW # OR CR)?" type the estimated number of analyses desired from this sample and (CR).
- 6.5.15. At the prompt "1 FILES ARE REQUIRED. START AT #?(NEW OR CR)?" type the next unused file number as noted in step 6.5.2. and (CR). (The ? will be the next unused file number as stored from Section 6.5.2).
- 6.5.16. At the prompt "DISK LABEL" check that the correct sample identifier is listed (as entered in section 6.5.12): if correct type a (CR); if incorrect type N (CR) and, when prompted, type the correct sample identifier.
- 6.5.17. The computer will now open the file on the Certified Operator's disk, and print the available mineral codes if accessing a mineral code routine. When the system is ready to perform an analysis it will prompt; "<WHITE> = WDS, <RED> = EDS, B=BLANK, U=UNBLANK, <CNTRL-G> = END" Pressing the white button will start a WDS analysis; pressing the red button will start an EDS acquisition, and typing a control-G will close the QUANT routine and return to the main TASK program.
- 6.5.18. Check that the standard you wish to analyze is in focus optically on the TV monitor and then press the white joystick button.
- 6.5.19. If using BA Definition file 110, the computer will ask "ENTER THE MINERAL CODE:". Type the three letter mineral code of your choice (see 6.5.17) and (CR).
- 6.5.20. If a Bence-Albee routine is being used, when the analysis is complete the computer will prompt "SAVE THIS ANALYSIS (Y/N)?" The Certified Operator will verify that the results are within acceptable limits of the listed

values for the standard analyzed. It is the Certified Operator's responsibility to define his or her acceptance criteria, however, if the calculated value is within \pm two sigma (the error printed to the right of the calculated weight percent is one sigma) of the listed values then the analysis is generally considered acceptable. If the above prompt is answered with a Y (CR), then the computer will prompt for a "TAG" for this point. The Operator may enter an identifying tag composed of a maximum of 23 characters. Caution: use no spaces or commas. (If the PRZ routine is used there will be no save/reject or tag option-all points will go to disk).

- 6.5.21. If, in the Certified Operator's judgement, one or more elements are not in calibration, then type a period (.) to return to the TASK prompt and calibrate. Individual elements are calibrated by typing (at the TASK8: prompt) "CAL XXX" (where XXX stands for the reference element, e.g., Si1) and (CR). The stage will drive to the selected standard coordinates and prompt the operator "HIT JOYSTICK #1 WHEN READY". Select a spot for calibration, focus optically, and push the white button on the joystick box.
- 6.5.22. Following calibration of one or more elements, standards should be re-run to assess the new calibration. If the Certified Operator deems the EMP system to be in calibration then he/she should type, (at the TASK8: prompt), SAV EL 110 (CR) or the number of the element table currently being used. This will save the current calibration to disk. Answer the following question prompts.
- "110 MULTIREF BA (DATE LAST SAVED) OK?" The question is asking if you intend to write over the old file.
 - If your answer is yes, type (CR). This will update the calibration on disk.
 - When prompted by the program, "ENTER LABEL," type 110 MULTIREF BA, (or the number of the element table being used), the current date, and then a (CR).
 - If you intend to store the current calibration under a new file number type N (CR) and, when prompted, type the new file number. The computer will check to determine if the new number is used. If not, a title will be requested. The title is limited to 23 characters. Use your last name, some identifier mnemonic, and the date. If the new number is already used consult with machine custodian for an unused file number.
- 6.5.23. When the Certified Operator deems the system to be calibrated then analysis results from the appropriate standards must be stored on disk and analysis of ER samples may proceed.
- 6.5.24. The Certified Operator may type a period (.) with no (CR) and return to the TASK prompt for execution of any one TASK command. At the completion of the TASK command there will be no prompt and the system will wait for a joystick button to be pressed (i.e. initiate a quantitative or qualitative

analysis) or for another period (.) to be typed. Refer to "SANDIA TASK8C" for more information regarding TASK commands.

6.6 Quantitative Unknown Analysis

- 6.6.1. Once appropriate standards have been run and the EMP System is deemed calibrated then an unknown sample may be analyzed. The following commands may be issues from 'within' a Quant routine by typing the period (.) for execution of one TASK command, or the Quant file may be closed out (control-G) and another fill opened for the unknown sample.
- 6.6.2. To insert a sample into the EMP for analysis, first ascertain that there is no sample currently resident in the EMP. Look at the position of the sample shuttle handle: if it is in the 3:00 o'clock position there is probably a sample in the chamber. If there appears to be a sample in the EMP refer to 6.7. If the shuttle handle is in the 12:00 o'clock position continue with this sequence.
- 6.6.3. Type CH (CR) (or if "within" a Quant routine, type period (.), wait for the TASK8: prompt, then type CH (CR)). This will move the stage to the sample change coordinates. Before proceeding compare the mechanical stage indicator positions with the sample change positions posted on the EMP. If the mechanical indicators are not correct, use the joystick to drive to the correct coordinates. Load the sample in the sample holder and screw the holder into the airlock with the handle in the 12 o'clock position.
- 6.6.4. Press the Pump-Down button and wait for the Valve Driving LED to come on. (If it does not come on consult with machine custodian.)
- 6.6.5. Once the Valve Driving LED is on, open the airlock lever counter-clockwise. While keeping the shuttle handle in the 12:00 o'clock position, push the shuttle handle all the way in, and then turn the shuttle handle to the right 90 degrees to the 3 o'clock position. Pull the shuttle handle out, still in the 3 o'clock position.
- 6.6.6. Close the airlock valve by rotating the airlock lever clockwise.
- 6.6.7. Press the Air Inlet button and wait about 3 seconds. Press the Off button.
- 6.6.8. Type a period (.) and wait for the TASK8: prompt. Then type GET POINT 46 (CR). This will move the stage to the approximate center of the thin section holder.
- 6.6.9. Analyze the unknown sample following sections 6.5.5-6.5.24 if opening a new file, or section 6.5.18-6.5.24 if using an existing file.

- 6.6.10. Standards should be run several times throughout an analysis session or whenever the Certified Operator questions the validity of an analysis.

6.7 Changing Samples

- 6.7.1. Type a period (.) and wait for the TASK8: prompt. Then type CH (CR). The stage will move to the sample change coordinates.
- 6.7.2. Verify that the mechanical indicators match the sample change coordinates listed in the microprobe logbook. If they do not, use the joystick controls to drive to the change coordinates.
- 6.7.3. Press the Airlock Pump Down button.
- 6.7.4. Wait until the valve driving LED is on.
- 6.7.5. Open the Airlock valve by rotating the airlock lever counter-clockwise.
- 6.7.6. Insert shuttle handle with the handle in the 3 o'clock position and when in completely, rotate the handle counter-clockwise to the 12 o'clock position.
- 6.7.7. Pull the sample holder handle out.
- 6.7.8. Close the Airlock valve by rotating the airlock lever clockwise.
- 6.7.9. Press the Air Inlet button and wait 3 seconds.
- 6.7.10. Press the Off button.
- 6.7.11. Unscrew the sample holder from the Airlock.
- 6.7.12. Change sample holders.
- 6.7.13. Screw the sample holder back into the Airlock
- 6.7.14. Repeat steps 6.7.3 to 6.7.8.

6.8 Shut Down of Microprobe

- 6.8.1. Remove the thin section from probe following steps in section 6.7.
- 6.8.2. If still in a QUANT routine, terminate it by typing CONTROL-G.
- 6.8.3. Put the probe in an idle mode.
 - 6.8.3.1. Type LOA SCHED 10 (CR). This will load schedule 10 and return the TASK8: prompt.

- 6.8.3.2. Type RUN BYBY (CR). This schedule will drive each spectrometer to a safe position (.50), and park it there, position the stage to the STANDARD FOCUS, and release the beam to an unregulated mode.
- 6.8.3.3. Wait until the absorbed current meter indicates the beam is on and then turn the Filament Temperature knob gently counter-clockwise to zero.
- 6.8.3.4. Release the Filament Power Supply button.
- 6.8.3.5. Turn off the TV camera adaptor.
- 6.8.3.6. Turn off the power switch on the TV Monitor.
- 6.8.3.7. Release the Light Microscope Illuminator button and toggle the switch below it to low.
- 6.8.3.8. Deactivate the High Vacuum System by flipping the toggle switch on the left front of the Vacuum Chassis to STANDBY.
- 6.8.3.9. Remove your floppy disk or diskette and place it in the storage box.
- 6.8.3.10. Power down the SyQuest if it is in use.
- 6.8.3.11. Turn the TN Monitor contrast down to the minimum.

6.9 Instrument Calibration

6.9.1 EDS Gain and Resolution

The gain on the EMP System EDS will be checked annually by following the energy calibration instructions in the Series II X-Ray Analyzer Operator's Manuals using copper energy lines. Tolerance is ± 20 eV. If the gain is out of tolerance, it will be the machine custodian's responsibility to calibrate it by running the gain calibration program as described in the Tracor Northern Manual. The resolution of the Series II EDS will be checked annually by measuring the full width at half maximum of the K alpha line from manganese. This is a check only for degradation of the detector; no calibration or adjustment is made.

6.9.2 Faraday Cup

The machine custodian will check the accuracy of the Faraday Cup annually with a 1.34 volt Mercury battery current source (1.34 volts is a physical constant for a Mercury battery). Acceptable values are 9.95×10^{-9} amperes, $\pm 1 \times 10^{-9}$ amperes. If the accuracy of the Faraday Cup is out of tolerance, it will be the machine custodian's responsibility to affix a tag stating "DO NOT USE" to the instrument and to repair the instrument so that it will be within the required tolerance (refer to Section 6.6 and 6.7 of TWS-QAS-QP-12.1).

6.10 Acceptance/Rejection Criteria

Quantitative X-ray analytical data must have acceptance limits set by operators. Some potential sources of uncertainty will be revealed by the inability to achieve an acceptable calibration as evidenced by running the certified standards as unknowns and comparing the calculated values to the listed standard values. Operators will accept or reject all quantitative data based on analyses of certified standards.

Special research needs may require deviations from this procedure. The operator must document any deviations in his/her ER logbook.

Following completion of EMP analytical investigation, samples will either be maintained in the custody of the Investigator or returned to the permanent sample storage room. Use the Chain-of-Custody/Request for Analysis form in SOP-01.04 to document custody and sample traceability.

7.0 REFERENCES

Documents referenced in this procedure are:

LANL-ER-SOP Section 1.0, General Instructions

LANL-ER-SOP-09-09, Certification of Standards for Electron Microanalysis

Cameca Model MBX Electron Probe/SEM Description, Use, Maintenance Manual, Cameca Instruments, Inc. Stanford, Connecticut

Series II X-Ray Analyzer Operator's Manuals, Noran Instruments, Inc. (formerly Tracor Northern, Inc.), Middleton, Wisconsin

Instruction Manual, Ladd Vacuum Evaporator, cat. no. 30000, 27 pgs. Ladd Research Industries, Inc., Burlington, Vermont

BA85: A Bence-Albee Oxide Analysis Routine with Mineral Code Capabilities, Sandia Report SAND90-1702

PRZ85: A Subroutined Quantitative Analysis Correction Procedure using the Bastin $q(rz)$ Technique. RFP 3215

SANDIA TASK8: A Subroutined Electron Microprobe Automation system, Sandia Report 2037, 1985

CONFIG8 A Configuration File Generator for SANDIA TASK8, Sandia Report 2035, 1985

The following procedure is from the Los Alamos National Laboratory Yucca Mountain Project Quality Assurance Manual in effect April, 1990.

TWS-ESS-DP-122, Preparation of Electron Microprobe Standard Mounts

8.0 RECORDS

- 8.1. Certified Operators record the following information in their ER notebooks: the ER sample number (cross-referenced to the sample identifiers used during analysis sessions), date, relevant instrument working parameters, any data files generated, records of standards used to calibrate elemental analyses, acceptance criteria, deviations, and additional information appropriate to interpreting results and to reproducing analyses. The Certified Operator will retain all printed output from analytical sessions with the EMP System.
- 8.2. The floppy disks and/or diskettes assigned to the Certified Operator are temporary storage for analytical data and will be kept in the EMP System room. It will be the Certified Operator's responsibility to edit, archive, and/or delete these files as needed.
- 8.3. All ER EMP System microanalysis data will be archived in a database on magnetic media.
- 8.4. The results of gain calibration, and Faraday cup and resolution checks, date, and name of the person performing the calibration and checks will be recorded in the ER EMP System logbook. A sticker will be placed on the Series II X-ray Analyzer console.
- 8.5. LANL notebooks, logbooks, EMP System data output, and other records are controlled in accordance with the procedures in Section 1.0, General Instructions.

9.0 ATTACHMENTS

N/A